

JP 61-296,099 A

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1505-90705

Translated from Japanese by the Ralph McElroy Translation Company  
910 West Avenue, Austin, Texas 78701 USA

JAPANESE PATENT OFFICE  
PATENT JOURNAL (A)  
KOKAI PATENT APPLICATION NO. SHO 61[1986]-296099

Int. Cl. <sup>4</sup> :	C 11 D 10/02 //C 11 D 17/00 (C 11 D 10/02 1:14 1:29 3:32 3:06)
Sequence No. for Office Use:	7144-4H
Filing No.:	Sho 60[1985]-137333
Filing Date:	June 24, 1985
Publication Date:	December 26, 1986
No. of Inventions:	1 (Total of 6 pages)
Examination Request:	Not filed

LAYER-SEPARATION TYPE LIQUID DETERGENT COMPOSITIONS

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[There are no amendments to this patent.]

### Claims

1. A layer-separation type liquid detergent composition, characterized by comprising at least one anionic surfactant such as a polyoxyalkyl ether sulfate or alkanesulfonate, an alkanolamide and 5-25 wt% of a condensed phosphate, where the weight ratio of the aforementioned anionic surfactant and alkanolamide (surfactant/alkanolamide) is 10/1 to 2/1.

2. The layer-separation type liquid detergent composition according to Claim 1, wherein the aforementioned polyoxyalkyl ether sulfate is expressed by general formula (1) below:



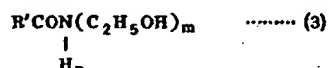
(where R denotes an alkyl group with an average carbon number of 8-18, *l* denotes a number with an average value of 1-10, M denotes an alkali metal ion, alkanolamine ion or alkaline earth metal ion).

3. The layer-separation type liquid detergent composition according to Claim 1, wherein the aforementioned alkanesulfonate is expressed by general formula (2) below:



(where PS denotes a linear or branching alkylsulfonate acid group with an average carbon number of 10-18, and M denotes an alkali metal or alkaline earth metal).

4. The layer-separation type liquid detergent composition according to any of Claims 1-3, wherein the aforementioned alkanolamide is a compound expressed by general formula (3) below:



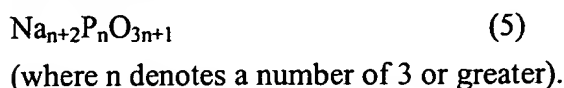
(where R' denotes an alkyl group with an average carbon number of 6-16, m denotes 1 or 2 and m+n = 2).

5. The layer-separation type liquid detergent composition according to any of Claims 1-4, wherein the aforementioned condensed phosphate is a sodium metaphosphate expressed by general formula (4) below:



(where n denotes a number of 3 or greater).

6. The layer-separation type liquid detergent composition according to any of Claims 1-4, wherein the aforementioned condensed phosphate is a sodium polyphosphate expressed by general formula (5) below:



#### Detailed explanation of the invention

The present invention relates to a layer-separation type liquid detergent composition that has excellent foaming capacity and storage stability.

#### Prior art

In the past, layer-separation type shampoos or detergents have been developed wherein, in order to increase the storage stability of the various contained components, the respective components separate into two layers without mixing when left in a stationary state. Examples of known compositions of this type are compositions that contain 2-ethylhexanoate higher alcohol esters (Japanese Kokoku Patent No. Sho 51[1976]-183), compositions that contain olive oil and squalane (Japanese Kokai Patent Application No. Sho 48[1973]-58007 and Japanese Kokai Patent Application No. Sho 48[1973]-10360), compositions that contain liquid paraffin and almond oil (Japanese Kokai Patent Application No. Sho 54[1979]-24908 and Japanese Kokai Patent Application No. Sho 54[1979]-25906) and other oily substances. These separation-type liquid detergent compositions are used after mixing the two layers by lightly shaking at the time of use. The oily substances separate when in a static state, thus improving storage stability.

However, these separation type compositions contain large amounts of the aforementioned oily substance, and thus have extremely poor foaming capacity, and are not appropriate for use as detergents.

On the other hand, also known is the use of two-layer separation type shampoos that are produced by blending hexametaphosphate followed by salting out (European Patent No. 116422). This shampoo has good foaming capacity, but has the disadvantage that separation degrades gradually with repeated shaking. Specifically, a cloudy layer is generated near the interface between the upper and lower layer, so that a clean separated state is not maintained in terms of external appearance.

#### Problems to be solved by the invention

The present invention has the objective of offering a layer-separation type liquid detergent composition that has excellent foaming performance and storage stability, while also providing clear and visually appealing separation, even with repeated shaking.

### Means to solve the problem

The layer-separation type liquid detergent composition of the present invention is characterized by comprising at least one anionic surfactant such as a polyoxyalkyl ether sulfate or alkanesulfonate, an alkanolamide and 5-25 wt% of a condensed phosphate, where the weight ratio of the aforementioned anionic surfactant and alkanolamide (surfactant/alkanolamide) is 10/1 to 2/1.

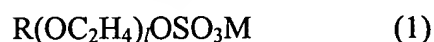
### Effect of the invention

With the layer-separation type liquid detergent composition of the present invention, layer separation is not affected by blending of the oil component, as described above, and thus the composition has superior foaming. In addition, even if shaking is repeatedly carried out during use, layer separation that is clean and visually appealing will be maintained, and a cloudy layer near the interface will not be produced. Consequently, the composition will have superior external appearance and high product value.

### Specific description of the invention

The layer-separation type liquid detergent composition of the present invention is described in detail below.

With regard to the anionic surfactant that is the first essential component of the composition of the present invention, various polyoxyethylene alkyl ether sulfates can be used, but the compounds expressed by general formula (1) below are particularly desirable:



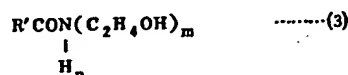
(where R denotes an alkyl group having an average carbon number of 8-18, with 10-14 being preferred, *l* denotes a number having an average value of 1-10, with 1-5 being preferred, and M denotes an alkali metal ion, alkanolamine ion or alkaline earth metal ion).

Specific examples of compounds represented by general formula (1) that can be cited include natural lauryl alcohol polyoxyethylene (n=3) sulfate ester, C<sub>11</sub>-C<sub>15</sub> aliphatic alcohol polyoxyethylene (n=3) sulfate esters synthesized by the oxo method, C<sub>12</sub>-C<sub>13</sub> aliphatic alcohol oxyethylene sulfate esters synthesized by the oxo method, C<sub>12</sub>-C<sub>13</sub> aliphatic alcohol sulfate esters and compounds wherein these sulfate ester moieties form sodium salts, potassium salts or alkanolamine salts. Among these compounds, polyoxyethylene (n=3) lauryl ether sulfate sodium salt is particularly desirable. In the present invention individual compounds may be used, or two or more may be used in conjunction.

On the other hand, examples of the alkanesulfonate that is the other anionic surfactant include alkanesulfonate alkali metal salts or alkaline earth metal salts having average carbon numbers of 10-18, with 14-18 being preferred. These alkanesulfonates can be obtained by

subjecting n-paraffin with a carbon number of 10-18 to sulfoxidation to produce an alkanesulfonic acid, which can then be neutralized with alkali.

The alkanolamide that is the second essential component of the composition of the present invention can be obtained by using fatty acid methyl ester or fatty acid ethyl ester as starting material, and employing a method wherein the material is heated for a comparatively short period of time at 120-250°C without using catalyst, a method wherein the aforementioned starting material is allowed to react using an alkali metal, hydroxide thereof or alcoholate thereof as catalyst, followed by separation or neutralization, or a method wherein sodium bicarbonate is used as catalyst. In particular, alkanolamides expressed by general formula (3) below are preferably used.



(where R' denotes an alkyl group having an average carbon number of 6-16, with 10-14 being preferred, m denotes 1 or 2, and m+n=2).

Specific examples include condensation products of diethanolamine and higher fatty acids such as coconut oil fatty acid, lauric acid and stearic acid. These compositions can be used individually, or two or more can be used in conjunction. Among these substances, coconut fatty acid diethanolamide is preferred.

One example of a condensed phosphate that is the third essential component of the composition of the present invention is a compound manufactured by a method involving subjecting a mixture of sodium phosphate or orthophosphoric acid and sodium hydroxide to thermal dehydration. Compounds expressed by general formula (4) below are particularly desirable.



(where n denotes a number of 3 or greater).

Specific examples of compounds represented by formula (4) include sodium trimetaphosphate, sodium tetrametaphosphate, sodium hexametaphosphate and sodium metaphosphate. These compounds can be used individually, or two or more can be used in conjunction.

Other examples of condensed phosphates that can be used are the sodium polyphosphates represented by general formula (5) below.



(where n denotes a number of 3 or greater).

Specific examples of compounds expressed by formula (5) include sodium tripolyphosphate and sodium tetrapolyphosphate. These compounds can be used individually, or two or more can be used in conjunction.

In adding the aforementioned three essential components to the layer-separation type liquid detergent composition of the present invention, it is necessary for the weight ratio of the anionic surfactant and alkanolamide (anionic surfactant/alkanolamide) to be 10/1 to 2/1, with 5/1 to 3/1 being preferred, and for the blending amount of condensed phosphate to be 5-25 wt% with respect to the entire composition, with 10-20 wt% being preferred. If the weight ratio exceeds 10/1, separation properties will be poor, and a clear interface will not be manifested between the upper layer and lower layer. On the other hand, if this ratio is less than 2/1, separation will be favorable, but the upper layer or lower layer will not be transparent, which is undesirable.

If the blending amount of condensed phosphate is less than 5 wt%, layer separation will not occur, whereas if this amount is greater than 25 wt%, then storage stability will be poor, and clouding will occur at low temperatures or high temperatures, so that the objective of the present invention cannot be attained.

Auxiliary additives that are commonly used can also be added as necessary to the detergent composition of the present invention, depending on the type and objectives of use. Examples of additives that can be cited include sodium chloride, Glauber's salt, solubilizers, BHT,  $\alpha$ -tocopherols and other antioxidants, ultraviolet absorbers, protein derivatives, animal and plant extracts, antimicrobial agents, dyes, fragrance, cationic polymers and other conditioning agents.

In addition, with the objective of adjusting detergent properties or foaming properties, other anionic surfactants, nonionic surfactants and amphoteric surfactants can also be added in ranges in which the separation properties are not compromised. In this case, examples of other surfactants include  $\alpha$ -olefin sulfonates, higher alcohol sulfate ester salts, polyoxyethylene higher alcohol sulfate esters or salts thereof, polyoxyethylene higher fatty acid esters or salts thereof, N-acyl acidic amino acid salts or basic amino acid salts, amine oxides, alcohol or alkylphenol ethylene oxide addition products, sugar alcohol fatty acid esters, carboxybetaines and sulfobetaines. These surfactants can be used individually, or two or more can be used.

The layer-separation type liquid detergent composition of the present invention can be used in a wide range of applications as a kitchen detergent, clothing detergent, shampoo or other liquid detergent.

Application examples and comparative examples of the present invention are presented below in order to more specifically describe the effects of the present invention.

The test methods used in each of the examples will be discussed prior to describing the examples.

(1) Foaming power

20 mL of a 6% aqueous solution of a prepared detergent composition (25°C) are collected in a 100 mL cylinder, and 0.2 g of liquid lanolin is added as an artificial soiling substance. The solution is then shaken 20 times in 10 seconds, and the foaming volume (mL) is measured after 1 min.

(2) Separation properties

The prepared detergent composition is left to stand for about 1 day, and a visual evaluation is made as to the distinctness of the separation of the two layers according to the following criteria:

O: Separated into two clear layers

Δ: No interface seen between the two separated layers

X: Two layers not separated

(3) Separation stability (separation properties subsequent to repeated shaking)

The prepared detergent composition is introduced into a transparent container, and after shaking up and down 10 times, the solution is left for 1 day and allowed to separate into two layers. This shaking-standing cycle is repeated 10 times, and a visual determination is then made as to whether a cloudy layer has appeared at the interface according to the following criteria.

O: No cloudy layer, clear separation between the two layers

Δ: Cloudy layer somewhat visible

X: Cloudy layer clearly visible

Application Examples 1-6

17 detergent compositions having the blend compositions shown in Table 1 were prepared, and the performance of these compositions was evaluated. The results are shown in the table. As is clear from the table, the compositions that contained the three essential components of the present invention (Application Examples 1-6) all exhibited superior performance, whereas the compositions that did not contain all three of the essential components of the present invention (Comparative Examples 1-9) and compositions in which the third essential component was present outside the range of the present invention (Comparative Examples 10 and 11) did not exhibit superior performance.



Table 1

③ 成分	① 実施例											1	2	3	4	5	6		
	② 比較例											1	2	3	4	5	6	10	11
④ 直鎖アルキルベンゼンスルホン酸ナトリウム (分子量 346)	20																		
⑤ ヤシ油アルコール硫酸エステルナトリウム (分子量 305)		20																	
⑥ C <sub>14</sub> -α-オレフィンスルホン酸ナトリウム			20																
⑦ N-アシルグルタミン酸トリエタノールアミン				20															
⑧ ポリオキシエチレンアルキルエーテル硫酸ナトリウム (平均EO付加モル数3、アルキル基C <sub>12</sub> /C <sub>13</sub> =1/1)				20		20	20	20	15	15						15	15	15	15
⑨ アルカンスルホン酸ナトリウム (分子量328、アルキル基C <sub>14</sub> ~C <sub>18</sub> )					20									15	15				
⑩ ヤシ油脂肪酸ジエタノールアミド										5	5	5	5	5	5	5	5	5	5
⑪ 塩化ナトリウム	15	15	15	15	15	15													
⑫ 硫酸マグネシウム						15													
⑬ ヘキサメタリン酸ナトリウム							15		15		15					5	25	3	30
⑭ テトラポリリン酸ナトリウム								15		15		15							
⑮ エタノール	3																		
⑯ 水	②0 残部																		
⑰ 起泡力 (mL)	60	80	70	60	50	47	50	50	50	70	70	68	68	70	70	70	70	70	70
⑱ 分離性	×	×	×	×	×	△	△	○	○	○	○	○	○	○	○	○	○	×	○
⑲ 分離安定性	△	△	△	△	△	×	×	×	×	○	○	○	○	○	○	○	○	△	×

- Key:
- Application Example
  - Comparative Example
  - Components
  - Linear sodium alkylbenzenesulfonate (molecular weight 346)
  - Coconut oil alcohol fatty acid ester sodium salt (molecular weight 305)
  - C<sub>14</sub>-α-olefinsulfonate sodium salt
  - N-Acylglutamate triethanolamine
  - Polyoxyethylene alkyl ether sulfate sodium salt (average 3 molar EO addition, C<sub>12</sub>/C<sub>13</sub> alkyl group = 1/1)
  - Sodium alkanesulfonate (molecular weight 328, alkyl groups C<sub>14</sub>-C<sub>18</sub>)
  - Coconut oil fatty acid diethanolamide
  - Sodium chloride
  - Magnesium sulfate
  - Sodium hexametaphosphate
  - Sodium tetrapolyphosphate
  - Ethanol
  - Water
  - Foaming power (mL)
  - Separation properties

- 19 Separation stability  
20 Remainder

### Application Examples 7-11

7 detergent compositions having the blend compositions shown in Table 2 were prepared, and the performance was evaluated. The results are shown in the table. As is clear from the table, the compositions that employed non-condensed phosphate as the third essential component (Comparative Examples 12, 13) had inferior performance, whereas the compositions that employed condensed phosphate (Application Examples 7-11) had superior characteristics.

Table 2

成分	① 実施例						
	7	8	9	10	11	12	13
② 比較例							
④ ポリオキシエチレンアルキルエーテル硫酸ナトリウム (平均EO付加モル数3、アルキル基C <sub>12</sub> /C <sub>13</sub> =1/1)	15	15	15	15	15	15	15
⑤ アルカンスルホン酸ナトリウム (分子量328、アルキル基C <sub>14</sub> ~C <sub>18</sub> )							15
⑥ ヤシ油脂肪酸ジエタノールアミド	1.5	7.5	5	5	5	5	
⑦ ヤシ油脂肪酸モノエタノールアミド							5
⑧ モノメタリン酸ナトリウム			15				
⑨ トリメタリン酸ナトリウム				15			
⑩ ヘキサメタリン酸ナトリウム	15	15					15
⑪ ポリリン酸ナトリウム					15		
⑬ テトラポリリン酸ナトリウム						15	
⑬ エタノール	← 3 →						
⑭ 水	← ⑮ 残部 →						
⑮ 起 泡 力 (ml)	70	70	55	70	55	70	67
⑯ 分 離 性	○	○	△	○	△	○	○
⑰ 分 離 安 定 性	○	○	×	○	×	○	○

- Key: 1 Application Example  
2 Comparative Example  
3 Components  
4 Polyoxyethylene alkyl ether sulfate sodium salt (average 3 molar EO addition, C<sub>12</sub>/C<sub>13</sub> alkyl group = 1/1)  
5 Sodium alkanesulfonate (molecular weight 328, C<sub>14</sub>-C<sub>18</sub> alkyl group)  
6 Coconut fatty acid diethanolamide  
7 Coconut fatty acid monoethanolamide

- 8 Sodium monometaphosphate
- 9 Sodium trimetaphosphate
- 10 Sodium hexametaphosphate
- 11 Sodium polyphosphate
- 12 Sodium tetrapolyphosphate
- 13 Ethanol
- 14 Water
- 15 Foaming power (mL)
- 16 Separation properties
- 17 Separation stability
- 18 Remainder

### Application Example 12

The shampoo composition having the blend composition shown in Table 3 was prepared, and the performance was evaluated. The results are shown in the table. As is clear from the table, the shampoo composition had excellent performance.

Table 3

① 成 分	実施例 12
② ポリオキシエチレンアルキルエーテル硫酸ナトリウム (平均EO付加モル数3、アルキル基C <sub>12</sub> )	10
③ C <sub>14</sub> - $\alpha$ -オレフィンスルホン酸ナトリウム	5
④ ヤシ油脂肪酸ジエタノールアミド	3
⑤ ヘキサメタリン酸ナトリウム	15
⑥ 第4級塩素含有セルロースエーテル (カチオン化密度0.0014、分子量12万)	0.2
⑦ ポリエーテル変性シリコーン	0.2
⑧ 殺菌剤(安息香酸ナトリウム)	1.0
⑨ 香 料	0.4
⑩ 色 素(黄色203号)	微量 ⑪
⑪ エタノール	3
⑫ 水	残 留 ⑫
⑬ 起 泡 力 (mL)	72
⑭ 分 離 性	○
⑮ 分 離 安 定 性	○

Key:	1	Component
	2	Polyoxyethylene alkyl ether sulfate sodium salt (average 3 molar EO addition, C <sub>12</sub> alkyl group)
	3	C <sub>14</sub> - $\alpha$ -olefinsulfonate sodium salt
	4	Coconut oil fatty acid diethanolamide
	5	Sodium hexametaphosphate
	6	Cellulose ether containing quaternary nitrogens (cationization density 0.0014, molecular weight 120,000)
	7	Polyether-modified silicone
	8	Antimicrobial agent (sodium benzoate)
	9	Fragrance
	10	Dye (Yellow 203)
	11	Ethanol
	12	Water
	13	Foaming power (mL)
	14	Separation properties
	15	Separation stability
	16	Application Example
	17	Trace amount
	18	Remainder